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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte SVEN LINDFORS

Appeal 2009-013982
Application 10/615,332
Technology Center 1700

Decided: March 17, 2010

Before CHARLES F. WARREN, TERRY J. OWENS, and
MARK NAGUMO, *Administrative Patent Judges*.

NAGUMO, *Administrative Patent Judge*.

DECISION ON APPEAL

A. Introduction[1, 2]

Sven Lindfors ("Lindfors") timely appeals under 35 U.S.C. § 134(a) from the final rejection[3] of claims 12-17, 19-26, and 30-34. We have jurisdiction under 35 U.S.C. § 6. We AFFIRM.

The subject matter on appeal relates to methods of depositing films on semiconductor wafers, particularly by atomic layer deposition ("ALD"). According to Lindfors, many of the reactants used in ALD are low-vapor pressure liquids that require heating to high temperatures to obtain significant vaporization. Once vaporized, the materials must be maintained at high temperatures to prevent condensation before they react with other components and form films on the intended substrate. Because the high temperatures can accelerate thermal decomposition of the low-vapor pressure liquids, and because the dose introduced to the reaction chamber must be controlled carefully, it is desirable to limit the amount of liquid that is vaporized. Moreover, because the reactants are typically mutually very reactive, even at room temperature, it is important to keep the different reactants confined to the reaction chamber, to introduce them separately, and to keep them apart from one another elsewhere in the apparatus.

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[1] Application 10/615,332, M*ethod and Apparatus for the Pulse-Wise Supply of a Vaporized Liquid Reactant*, filed 8 July 2003, claiming the benefit of a provisional application filed 12 July 2002. The specification is referred to as the "332 Specification," and is cited as "Spec." The real party in interest is listed as ASM International N.V. (Appeal Brief, filed 23 February 2009 ("Br."), 3.)

[2] The oral argument scheduled for 11 March 2010 was waived.

[3] Office action mailed 15 July 2008 ("Final Rejection"; cited as "FR").

To accomplish these goals, the two or more reactants are introduced in alternating pulses to the reaction chamber. The production of alternating pulses involves sophisticated schemes such as “inert gas valving,” in which inert gases are used to create “inert gas diffusion barriers” by establishing higher pressures in certain gas lines of the apparatus to keep gases in lower pressure regions from invading the higher pressure regions.

Lindfors claims a method in which a liquid precursor, typically held at ambient temperature in a storage chamber, is introduced to a vaporization chamber at a higher temperature. The vapor is then introduced to a reaction chamber, in alternating pulses with another reactant, to deposit a film on a substrate. Unevaporated liquid in the evaporating chamber is then returned to the storage chamber, whence it can be used in a subsequent vaporization and reaction-deposition cycle.

Representative Claim 12 is reproduced from the Claims Appendix to the Principal Brief on Appeal, with labels to Figure 1 of Lindfors’s supporting 332 Specification (reproduced below at 7) added to illustrate—but not to limit—the claimed invention:

> 12. A method for providing vapor phase reactant from solid or liquid source, comprising:
>
> supplying a liquid comprising a precursor from a storage container [100] to a vaporization chamber [312];
>
> maintaining the vaporization chamber [312] at a higher temperature [T2] than the storage container [100];
>
> vaporizing the precursor in the vaporization chamber;
>
> transporting the vaporized precursor to a reaction chamber [410];

> conducting a vapor deposition process using the vaporized precursor in the reaction chamber;
>
> draining unvaporized liquid from the vaporization chamber [310] after conducting the vapor deposition process without opening the vaporization chamber;
>
> *returning the unvaporized liquid to the storage container* [100]; and
>
> returning the unvaporized liquid to the vaporization chamber
>
> wherein transporting comprises
>
>> supplying pulses of the vaporized precursor to the reaction chamber alternatingly with pulses of at least one other precursor and
>>
>> stopping and allowing flow of the vaporized precursor from the vaporization chamber to the reaction chamber with an inert gas diffusion barrier and wherein stopping and allowing flow with an inert gas diffusion barrier comprises
>>
>>> controlling valves for an inert gas flow outside of a hot zone accommodating the vaporization chamber.

(Claims App., Br. 17; indentation, paragraphing, emphasis, and bracketed labels added.)[4]

The Examiner has maintained the following grounds of rejection:[5]

> A. Claims 12-14, 19-23, 26, and 30 stand rejected under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami,[6] Modisette,[7] and Bondestram.[8]

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[4] Labels are shown in bold font throughout, for clarity, regardless of their presentation in the original document.

[5] Examiner's Answer mailed 28 April 2009. ("Ans.").

B. Claims 15 and 16 stand rejected under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Kaloyeros.[9]

C. Claims 17, 31, and 32 stand rejected under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, Kaloyeros, and Sturm.[10]

D. Claims 24 and 25 stand rejected under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Van Buskirk.[11]

E. Claims 33 and 34 stand rejected under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Gauthier.[12]

Lindfors contends there is no reason in the references to modify the structure described by Murakami and to use the modified structure to return liquid reactants from evaporator 8 to reservoir 4. (Br. 12.) According to Lindfors, Murakami is concerned with reactants clogging the liquid supply lines, and therefore teaches away from returning reactants to the reservoir. (Br. 11, first full para.) Lindfors argues that Modisette does not cure this

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6 Seishi Murakami and Tatsuo Hatano, *Liquid Material Supply Apparatus and Method*, U.S. Patent 6,126,994 (2000).

7 Jerry L. Modisette and Otto F. Schkade, *Vapor Recovery System and Method*, U.S. Patent 3,981,156 (1976).

8 Niklas Bondestram and Menso Hendriks, *Active Pulse Monitoring in a Chemical Reactor*, U.S. Patent 7,063,981 B2 (2006) based on an application filed 30 January 2002.

9 Alain E. Kaloyeros et al., U.S. Patent 5,476,409 (1994).

10 Edward A. Sturm et al., U.S. Patent 6,178,925 B1 (2001).

11 Peter C. Van Buskirk et al., U.S. Patent 5,882,416 (1999).

12 Scott Gautier, U.S. Patent 6,007,330 (1999).

deficiency because Modisette teaches condensing vapor from a storage tank by processes, presumably in the presence of air, that are returned to the storage tank. Because the contact with air is the specific problem that Murakami seeks to avoid, Lindfors argues, "the combination [with Murakami] does not work." (Br. 13, first para.)

The Examiner maintains that it would have been obvious to adapt the liquid delivery system described by Murakami to perform the ALD process described by Bondestram. (FR 6.) According to the Examiner, the difference between the claimed process and such a process is that Murakami directs unvaporized low-vapor pressure liquid remaining in the evaporator to a cold trap, rather than recycling the unevaporated liquid to the initial storage container. (*Id.* at 5.) Modisette, in the Examiner's view, shows that the re-use of materials is well-known in the chemical arts. (*Id.*) Moreover, according to the Examiner, the structures described by Murakami are capable of being used to perform the claimed method. (*Id.* at 2.) Thus, the Examiner concludes, it would have been obvious to modify the apparatus described by Murakami to return the unevaporated low vapor pressure liquid to the reservoir, while performing an ALD process as described by Bondestram. (*Id.* at 6.)

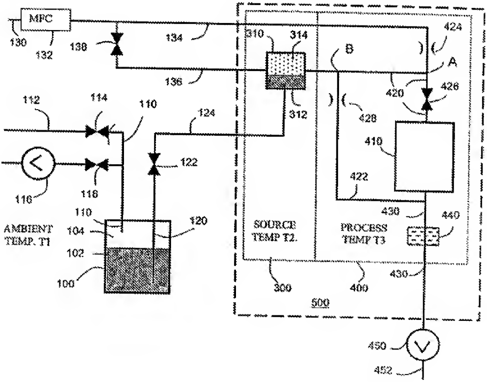
The dispositive issues in this case are whether Lindfors has shown that Murakami teaches away from recycling the unevaporated liquid left in evaporator 8; and whether the Examiner's reliance on Modisette is faulty.

B. Findings of Fact

Findings of fact throughout this Opinion are supported by a preponderance of the evidence of record.

The 332 Specification

The disputed portions of the appealed method are readily understood with reference to Figure 1 from the 332 Specification, which is reproduced below:



{Figure 1 is said to show an apparatus}

In the claimed process, storage container 100 is charged with liquid reactant 102. (Spec. 8, ¶ [0025].) The remainder of the container 104 is filled with inert gas via feed line 110. Upon pressuring storage container 100 with inert gas, liquid reactant 312 is advanced into

vaporization chamber 310 via transfer line 124. (*Id.*) Vaporization chamber 312 is held in hot zone 300 at temperature T2, which is higher than the ambient temperature T1 of the storage container. (*Id.* at ¶ [0026].) Vaporized reactant 314 collects above the unvaporized liquid 312, and is introduced to reaction chamber 410 in hot zone 400 by a pulse of inert gas passed through pulse valve 138 in inert gas supply line 136. (*Id.* at 9, ¶ [0029]-10, ¶ [0030].) Details of the inert gas diffusion barrier are not necessary to understanding this appeal. Similarly, the mechanisms for generating the alternating pulses of the other reactant, which are not shown in the Figures, are not necessary to resolve this appeal.

According to the 332 Specification, vaporization chamber 310 can be drained at any suitable time (Spec. 9, ¶ [0028]) by setting appropriate valves and raising the pressure in vaporization chamber 310 above the pressure in storage chamber 100, which may be evacuated by vacuum pump 116 (*Id.* at 8-9, ¶ [0027]).

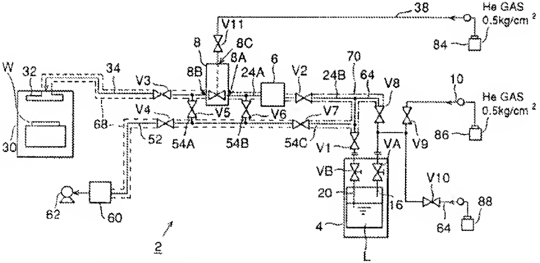
<u>Murakami</u>

Murakami describes an apparatus and method for providing a low vapor pressure liquid material via an evaporation chamber to a film deposition chamber. (Murakami, Abstract; col. 2, ll. 34-52.) In Murakami's words, "[t]he deposition chamber 30 is applicable to any deposition apparatus." (*Id.* at col. 4, ll. 38-39.) Murakami does not mention atomic layer deposition processes or apparatuses.

According to Murakami, the liquid materials used for making films typically have low vapor pressures and are very reactive and are readily

oxidized upon contact with air. (Murakami, col. 1, ll. 43-50.) Heating is said to provide the needed amount of vapor, but also to cause thermal decomposition problems. Moreover, reaction of residual liquid materials in pipes and connections with oxygen is said to "produce solid byproducts on replacement of liquid tanks," which clogs those pipes and connections. (*Id.* at col. 2, ll. 11-13.) Yet another problem is said to arise because the amount of liquid needed to provide the desired mount of gas is very small, making it "very difficult to supply stably and accurately the low vapor pressure liquid materials from the liquid tank to the deposition chamber." (*Id.* at col. 2, ll. 23-25.)

Murakami provides an apparatus, shown in Figure 1, reproduced below, and processes of using the apparatus, that are said to address these problems.



{Murakami Figure 1 is said to show an apparatus}

Low vapor pressure liquid L in reservoir 4 is moved, under pressure of helium gas provided via line 10, through discharge nozzle 20 and lines 24B and 24A to evaporator 8. (Murakami, col. 4, ll. 6-32.) The transfer lines and

the evaporation chamber are heated by heaters 68 and 70. (*Id.* at col. 5, ll. 56-67.) The liquid is vaporized, and the vapor then moved to reactor 30 via line 34 under helium pressure provided via line 38. (*Id.* at col. 4, ll. 34-38.)

According to Murakami, "evaporator 8 of this type has a dead space 50[13] unavoidably formed by its structure." (Murakami, col. 5, ll. 10-11.) As a result, residual liquid collets in the dead space 50, and when the next deposition cycle starts, the residual liquid is vaporized and deposited on the substrate, in addition to the liquid transferred deliberately from reservoir 4. As a result, too much material is provided, and proper films cannot be deposited on the wafers. (*Id.* at ll. 15-21.) To solve this problem, Murakami provides discharge passage 54A, which permits the residual liquid to be discharged via line 52 to cold trap 60. (*Id.* at ll. 26-40 and at col. 7, ll. 47-59.) In Murakami's words, "before a liquid material for deposition is actually flowed and fed, residual liquid material in the evaporator 8, etc. can be expelled." (*Id.* at ll. 47-50.) Moreover, Murakami teaches that "[t]he pre-expelling operation need not be conducted immediately before a deposition, but may be conducted immediately after a preceding deposition is over." (*Id.* at ll. 56-58.) According to Murakami, "[o]ccurrence of overshoots of a liquid material supply amount can be precluded in deposition, whereby the deposition material can be fed stably and accurately," resulting in higher quality films. (*Id.* at col. 10, ll. 45-50.)

[13] Dead space 50 is shown in Figure 2, but not reproduced here.

Murakami provides additional discharge passages 54B and 54C to remove of low vapor pressure liquid from the transfer lines via inert gas pressure and solvent washes when reservoir 4 needs to be changed. (Murakami at col. 5, ll. 26-40 and at col. 7, l. 50 to col. 9, l. 21.) Murakami explains the advantages of these features as follows:

> liquid material in the passages can be substantially perfectly expelled *without contact of the liquid material with air* for maintenance of the respective members and replacement of the reservoir. Accordingly production of byproducts by oxidation of the liquid material, and clogging of the passages with the byproducts can be precluded. Liquid material supply can be stable.

(*Id.* at col. 10, ll. 52-58; emphasis added.)

C. Discussion

As the Appellant, Lindfors bears the procedural burden of showing harmful error in the Examiner's rejections. *See, e.g., Shinseki v. Sanders*, 129 S. Ct. 1696, 1706 (2009) ("the burden of showing that an error is harmful normally falls upon the party attacking the agency's determination.").

Initially, we note that Lindfors has expressly waived argument in this appeal regarding the fitness of Bondestram as prior art in the rejection under § 103. Although Lindfors reserves the option of removing Bondestram by swearing behind or "asserting common co-ownership under 35 U.S.C. § 103(c)," Lindfors states that "we do not need to remove Bondestram at this time in view of the arguments in the body of the appeal brief." (Br. 12 n.1.) Thus, Lindfors concedes that Bondestram is, on the present record, properly applied as a reference under § 103. We note further

that Lindstrom has not advanced any substantive arguments regarding the teachings of Bondestram or the propriety of combining those teachings with the teachings of Murakami or Modisette, save that Bondestram does not cure the alleged defects of those references. (*Id.* at 12, 14.) Thus, Lindfors has also waived substantive argument regarding Bondestram. Finally, with regard to each of the obviousness rejections relying in part Kaloyeros (*id.* at 14), Sturm (*id.* at 15), van Buskirk (*id.*), or Gauthier (*id.* at 16), Lindfors states that "[t]he Appellant neither admits nor denies these allegations by the Examiner." (Br. 14, 15, and 16.) Rather, Lindfors merely asserts that the additional references do not remedy the deficiencies of the rejection of independent claim 12. Thus, Lindfors has also waived all substantive arguments regarding the additional references. Accordingly, all claims stand or fall with claim 12.

Lindfors' principal argument, that Murakami "teaches away from returning reactants to the liquid reservoir" (Br. 12, second full para.), is without merit. The Federal Circuit has explained that "[a] reference may be said to teach away when a person of ordinary skill, upon reading the reference . . . would be led in a direction divergent from the path that was taken by the applicant." *In re Gurley*, 27 F.3d 551, 553 (Fed. Cir. 1994). There is no indication in Murakami that contact of the highly reactive low vapor pressure liquid with oxygen is a problem in the "pre-expelling" step of removing the residual low vapor pressure liquid from the evaporator. It is clear from the disclosure that the evaporator and the transfer lines attached to it are maintained in an oxygen-free state during use. The passages that Lindfors cites relate to the desirability of preventing contact of the low vapor

pressure liquid with oxygen during exchange of an empty reservoir 4 for a full reservoir. The purpose of the helium-driven liquid expelling operations and the purge liquid operations described in columns 8-9 is precisely to insure maintenance of oxygen-free conditions in the apparatus during such an exchange. Thus, Murakami cannot be said to teach away from recycling the unevaporated low vapor pressure liquid in the absence of oxygen.

The critical differences, according to the Examiner, between the teachings of Murakami and the claimed process are the inert gas valving steps (which are associated with Atomic Layer Deposition processes), for which the Examiner relies on Bondestram, and the recycling of the low vapor pressure liquid, for which the Examiner relies on Modisette for evidence supporting motivation. As already noted, Lindfors has not argued error in the Examiner's reliance on Bondestram.

Lindfors' criticisms of the Examiner's reliance on Modisette are not well taken. We understand the Examiner to have relied on Modisette as evidence that recycling materials is a well known procedure in chemical processing. Modisette's illustration of the recovery of a solvent, and return of the solvent to the reservoir from whence it came, stands as evidence that a person having ordinary skill in the chemical arts would have been motivated to recycle a reagent, such as the unevaporated liquid collected by Murakami, in order to conserve materials. Such a person would have taken precautions to avoid known degradative conditions, such as exposure to oxygen, as a matter of routine. Thus, the Examiner's reliance on Modisette was not harmful, as a chemist of ordinary skill would have adopted the general teaching of recycling, but not the specific conditions described by Modisette.

Similarly, Lindfors has not shown harmful error in the Examiner's suggestion (FR 5, Ans. 5) that the valve VB and nozzle 20 could be used to return the low vapor pressure liquid to reservoir 4. Lindfors has not directed our attention to any credible evidence that the skilled reactor designer would not have been able to add components or to adopt existing components to perform the obvious recycling step.

We therefore AFFIRM the Examiner's rejections.

D. Order

We AFFIRM the rejection of claims 12-14, 19-23, 26, and 30 under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, and Bondestram.

We AFFIRM the rejection of claims 15 and 16 under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Kaloyeros.[14]

We AFFIRM the rejection of claims 17, 31, and 32 under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, Kaloyeros, and Sturm.[15]

We AFFIRM the rejection of claims 24 and 25 under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Van Buskirk.[16]

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[14] Alain E. Kaloyeros et al., U.S. Patent 5,476,409 (1994).

[15] Edward A. Sturm et al., U.S. Patent 6,178,925 B1 (2001).

[16] Peter C. Van Buskirk et al., U.S. Patent 5,882,416 (1999).

We AFFIRM the rejection of claims 33 and 34 under 35 U.S.C. § 103(a) in view of the combined teachings of Murakami, Modisette, Bondestram, and Gauthier.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a)(1).

<u>AFFIRMED</u>

kmm

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